ESRF	**Experiment title: Diffraction studies of grain growth kinetics in Nanocrystalline and amorphous Chromium.**	**Experiment number**: ME 378
Beamline: ID-31	**Date of experiment**: 28/06 – 01/07/2002	**Date of report**: 28/11/2002
Shifts: 9	**Local contact(s)**: Andy Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

R. Przeniosło (*)

ILL Grenoble and Institute of Experimental Physics, Warsaw University, Poland

G. Chojnowski(*), W. Sławinski (*), I. Sosnowska

Institute of Experimental Physics, Warsaw University, Poland

M. Bukowski (*) and R. Hempelmann

Inst. of Physical Chemistry, University of Saarbrücken, Germany

Report:

The grain growth in nanocrystalline (n-Cr) and amorphous (am-Cr) Chromium was studied by using high resolution SR diffraction at beamline ID-31. The initial n-Cr and am-Cr samples were previously characterized by X-ray diffraction, neutron diffraction and small-angle neutron scattering (SANS) [1,2]. These studies have shown that the n-Cr material has the same bcc structure as polycrystalline Cr, and its volume-averaged grain size is 27 nm. The large scale microstructure studies by SANS have shown density correlations of fractal type with surface fractal dimensions of 2.24(3) for n-Cr and 2.22(3) for am-Cr [1]. The neutron diffraction studies of n-Cr have shown a magnetic ordering which is different than in polycrystalline Cr [2].

The n-Cr and am-Cr samples were annealed at several temperatures ranging from 300°C to 700°C. The SR diffraction patterns were measured repeatedly during annealing with time

intervals of the order of one minute. The SR wavelength used was λ=0.24990Å. The time dependence of the FWHM of the strongest (110) Bragg peak of n-Cr is shown in Fig.1.

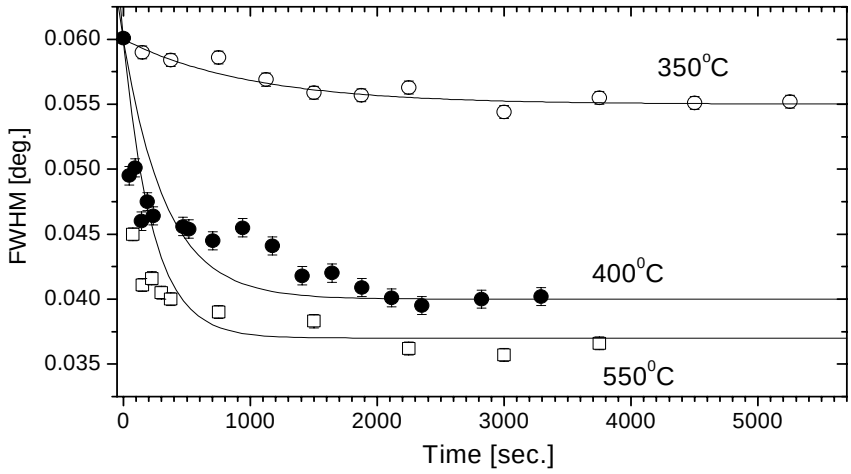

Fig.1 Time dependence of the FWHM of the (110) Bragg peak of n-Cr measured during annealing at three different temperatures (points). The lines are shown to guide-the-eye.

One can see that for annealing at 350°C the FWHM changes are small and they occur relatively slow. At higher annealing temperatures of 400°C and 550°C the reduction of the FWHM occurs faster but after some time the FWHM tends to a saturation value. This is an experimental evidence for the grain growth and/or strain reduction process in n-Cr due to annealing. First one should note that the FWHM values shown in Fig.1 are about 6-10 times larger that the instrumental resolution of ID-31. The observed broad FWHM is therefore mainly due to the reduced particle size and the strains. At the short wavelength used in the present experiment the FWHM of the (110) peak is mainly due to the the particle size. One can make some approximate calculation of the average particle size by using the Scherrer formula. One obtains 24 nm for the initial n-Cr while the saturation values after annealing at 400°C and 550°C are 36 nm and 40 nm respectively. Even at the highest annealing temperature 700°C, the saturation value of FWHM is 0.03° what corresponds to 48 nm only. This behavior of n-Cr differs very much from the behaviour of n-Fe, where similar experiments at 300-500°C have shown a grain growth up to 200-400 nm [3], i.e. 4-5 times more than in the present case. The reason for such a different behaviour is at present not clear. The diffraction patterns measured performed during annealing of am-Cr at 200 °C are shown

in Fig. 2. One can see that with annealing time the contribution of the amorphous phase decreases at the expense of the crystalline phase.

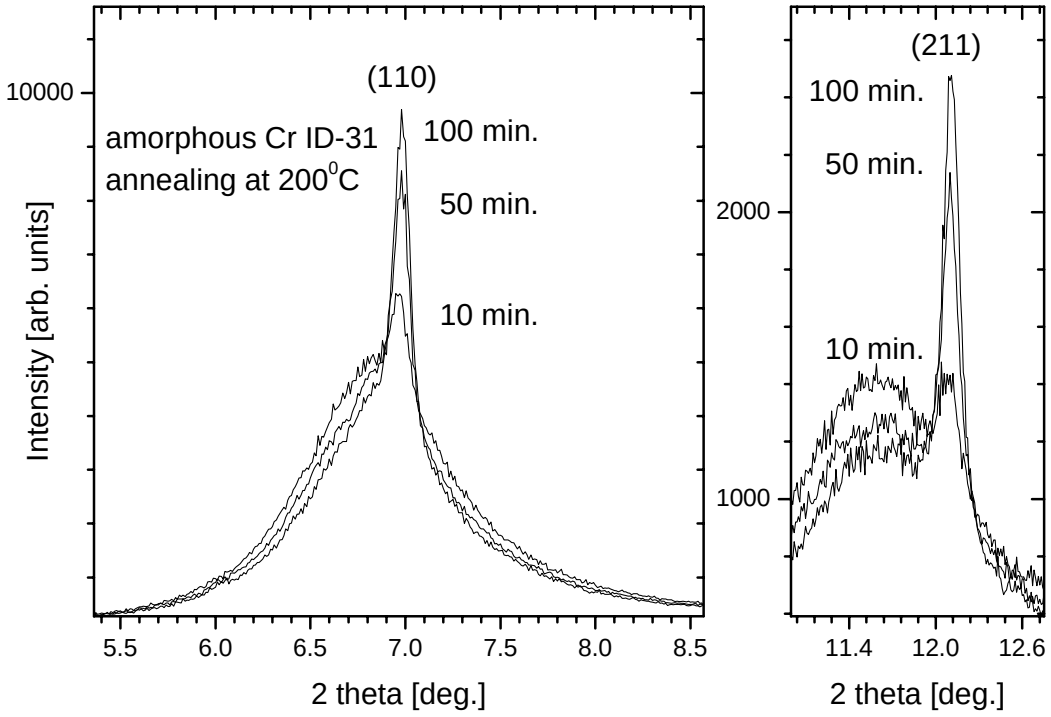

Fig.2 Time dependence of the diffraction pattern of am-Cr measured during annealing at 200ºC. After 100 minutes the crystallization process stops.

After an annealing time of 100 min. the crystallization process stops and the diffraction pattern do not change any more. It is also important to note that the final crystalline state of Cr is in fact also some kind of nanocrystal. It is difficult to estimate the FWHM of the crystalline Bragg peaks emerging on top of broad amorphous humps. It can be however estimated that the grain size is of the order of 20-30 nm. Further data analysis is in progress.

This work was supported by the European Community Marie Curie Fellowship performed at I.L.L. by one of us (RP) under contract HPMF-CT-2000-01002. This work was partially supported by Committee for Scientific Research (Poland).

References

[1] R. Przeniosło, J. Wagner, H. Natter, R. Hempelmann and W. Wagner, J. Alloys and Compounds **328** (2001) 259.

[2] R. Przeniosło, I. Sosnowska, G. Rousse and R. Hempelmann, Phys Rev. **B66** (2002) 014404.

[3] H. Natter, M. Schmelzer, M.-S. Löffler, C.E. Krill, A. Fitch and R. Hempelmann, J. Phys. Chem **B104** (2000) 2467.

[4] C. E. Krill III, L. Helfen, D. Michels, H. Natter, A. Fitch, O. Masson, and R. Birringer, Phys. Rev. Lett. **86** (2001) 842.